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IS 12795 (1989): Linear alkyl benzene [CHD 25: Soaps and other Surface Active Agents]

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IS 12795 : 1989
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Indian Standard

LINEAR ALKYL BENZENE — SPECIFICATION

भारतीय मानक

रैखिक ऐल्किल बेंजीन — विशिष्टि

(First Reprint DECEMBER 1992)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

***September* 1990** **Price Group 3**

Soaps and Other Surface Active Agents Sectional Committee, CDC 35

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 30 August 1989, after the draft finalized by the Soaps and Other Surface Active Agents Sectional Committee had been approved by the Chemical Division Council.

Linear alkyl benzene (LAB) is one of the materials used as a surfactant in synthetic detergents. It is a biodegradable material and is therefore finding greater acceptability since it has been found that effluent water containing detergents based on non-biodegradable raw materials could not be rendered harmless even after rigorous water treatment.

The manufacture of LAB has started in a big way in India and today India is self-sufficient in LAB.

Linear alkyl benzene is an important raw material for manufacture of household and industrial synthetic detergent powders, tablets and liquid detergents. The limitations of soaps for use in hard water areas has led to the development of synthetic detergents industry. The shortage of oils and fats, the main raw materials for the production of soaps and increase in their demand for edible purposes has further helped the growth of synthetic detergent industry in India.

Informulation of this standard considerable assistance has been derived from Indian Petrochemicals Corporation Limited and Universal Oil Products, USA.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values *(revised)'*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 2 AUGUST 1999
TO
IS 12795 : 1989 LINEAR ALKYL BENZENE — SPECIFICATION

[*Page* 2, *Table* 1 (*see also Amendment No.* 1)] — Substitute Table given on page 2 for the existing table.

(*Page* 2, *Annex* B) — Delete and renumber the subsequent Annexes as B to E from C to F.

(*Page* 6, *Annex* E) — Insert the following Annex F at the end:

'ANNEX F
(Table 1)

METHOD FOR SULPHONATION OF LINEAR ALKYL BENZENE

F-0 OUTLINE OF THE METHOD

F-0.1 Take 240 g of linear alkyl benzene into a one litre capacity three-neck flask. Add 240 g of 15-20 percent oleum to it slowly from the funnel attached to the flask, while constantly stirring. Maintain the temperature of the mass between 30°C and 35°C by cooling the flask in a water bath. After the addition of oleum is over, continue stirring for a further period of three hours maintaining the temperature of the water bath at 45-50°C. Add 40 ml water and stirr thoroughly. Transfer the material to a separatory funnel and allow it to separate into two layers. The separation takes 4-5 hours. Discard the lower layer of spent acid. Take the upper layer, which is sulphonk add for the ready biodegradability analysis.'

Table 1 Requirements for Linear Alkyl Benzene

(*Clauses* 4.2 *and* 7.1)

Sl No.	Characteristic	Requirement	Method of Test, Refer to			
			Part of IS 1448	Cl No. of IS 286	Annex	IS No.
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Colour					
	a) Saybolt, *Min*	30	—	—	A	–
	b) In 5" cell on Lovibond scale, expressed as *Y + 5R, Max*	0.5	–	—	A	–
ii)	Specific gravity at 15°/15°C	0.858-0.868	P 32	—	—	—
iii)	Motsture, ppm, *Max*	100	—	4	—	—
iv)	Doctor test	Negative	P 19	–	—	—
v)	Flash Point (Closed cup)°C, *Min*	130	P 21	—	—	—
vi)	Bromine Number, *Max*	0.05	P 44	—	—	—
vii)	Ready biodegradability of sulphonate*	To pass the test	—	—	—	13933
viii)	Distillation, 95 percent recovery at °C, *Max*	320	P 18	—		—
ix)	Molecular weight	236-246	—	—	B	—
x)	Paraffins, percent by mass, *Max*	0.2	—	—	C	—
xi)	Sulphonability, percent by mass, *Min*	98.0	—	—	D	—
xii)	Chain-length distribution, percent		–	—	B	
	a) Less than C_{10}, *Max*	0.5				
	b) C_{10}, *Max*	15.0				
	c) $C_{10} + C_{11}$	30.0-55.0				
	d) C_{12}	25.0-50.0				
	e) C_{13}, *Max*	30.00				
	f) C_{14}, *Max*	5.0				

*Linear alkyl benzene is to be sulphonated before carrying out ready biodegradability test, as given in Annex F

(CHD 25)

AMENDMENT NO. 1 FEBRUARY 1992
TO
IS 12795 : 1989 LINEAR ALKYL BENZENE — SPECIFICATION

[*Page* 2, *Table* 1, *SI No.* (v), *col* 3] — Substitute '130°C' *for* '1.30'.

[*Page* 3, *clause* B-2.2, *last line*] — Substitute '11' *for* '1.01 g'.

CHD 025)

Reprography Unit, BIS, New Delhi, India

Indian Standard

LINEAR ALKYL BENZENE – SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for linear alkyl benzene.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
IS 286 : 1978	Methods of sampling and test for soaps
IS 1070 : 1977	Water for general laboratory use
IS 1447 : 1960	Methods of sampling of petroleum and its products
IS 1448 [P : 13] : 1960	Methods of test for petroleum and its products, [P : 13] Colour by Lovibond tintometer
IS 1448 [P : 14] : 1960	Methods of test for petroleum and its products, [P : 14] Colour by Saybolt chromometer
IS 1448 [P : 18] : 1967	Methods of test for petroleum and its products, [P : 18] Distillation
IS 1448 [P : 19] : 1960	Methods of test for petroleum and its products, [P : 19] Doctor test
IS 1448 [P : 21] : 1970	Methods of test for petroleum and its products, [P : 21] Flash point
IS 1448 [P : 32] : 1972	Methods of test for petroleum and its products, [P : 32] Density and relative density
IS 1448 [P : 44] : 1983	Methods of test for petroleum and its products, [P : 44] Bromine number by electrometric titration method
IS 8401 : 1977	Specification for alkyl benzene sulphuric acid (acid slurry)
IS 7597 : 1974	Glossary of terms related to surface active agents

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given in IS 7597 : 1974 shall apply.

4 REQUIREMENT

4.1 Description

Linear alkyl benzene shall be a clear, colourless liquid with a characteristic odour.

4.2 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in moist-free mild steel tanks or barrels or as agreed to between the purchaser and the supplier.

5.2 Marking

The container shall be securely closed and marked with the following particulars:

a) Name of the material;

b) Indication of source of manufacture;

c) Net mass of the material;

d) Batch number or lot number in code or otherwise; and

e) Month and year of manufacture.

6 SAMPLING

6.1 The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in IS 1447 : 1960.

7 TESTS

7.1 Tests shall be carried out as prescribed in relevant parts of IS 1448, IS 286 : 1978 and Annexes A to F of this standard. References to the relevant methods of IS 1448 and relevant clauses of IS 286 : 1978 and Annexes A to F are given in col 4, 5 and 6 respectively of Table 1.

7.2 Quality of Reagents

Unless specified otherwise pure chemicals and distilled water (*see* IS 1070 : 1977) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of the analysis.

Table 1 Requirements for Linear Alkyl Benzene

(*Clauses* 4.2 *and* 7.1)

SI No.	Characteristic	Requirement	Method of Test, Refer to		
			Part of IS 1448	Clause No. of IS 286 : 1978	Annex
(1)	(2)	(3)	(4)	(5)	(6)
i)	Colour				
	a) Saybolt, *Min*	30	—	–	A
	b) In 5¼ inch cell on Lovibond scale, expressed as $Y + 5R$, *Max*	0.5			
ii)	Specific gravity at 15°/15°C	0.858-0.868	P 32	—	–
iii)	Moisture, ppm, *Max*	100	—	4	—
iv)	Doctor test	Negative	P 19	–	—
v)	Flash point (closed cup) °C, *Min*	1.30	P 21	—	—
vi)	Bromine No., *Max*	0.05	P 44	—	—
vii)	Biodegradability of sulphonate, percent, *Min*	92.0	—	—	B
viii)	Distillation, 95 percent recovery at °C, *Max*	320	P 18	—	–
ix)	Molecular weight	236-246	—	—	C
x)	Paraffins, percent by mass, *Max*	0.2	—	—	D
xi)	Sulphonability, percent by mass, *Min*	98.0	—	—	E
xii)	Chainlength distribution, percent				F
	Below C_{10}, *Max*	0.5			
	C_{10}, *Max*	15.0			
	C_{10}, + C_{11}	30.0-55.0			
	C_{12}	25.0-50.0			
	C_{12}, *Max*	30.00			
	C_{14}, *Max*	5.0			

ANNEX A

[*Table* 1, *Sl No.* (i) and *Clause* 7.1]

DETERMINATION OF COLOUR

A-0 GENERAL

A-0.1 The colour of the material can be determined by either Saybolt chromometer method or Lovibond tintometer method. However, in case of any dispute, Saybolt chromometer method shall be the referee method.

A-1 SAYBOLT CHROMOMETER METHOD

A-1.1 The colour shall be determined by the method specified in IS 1448 [P : 14] : 1960.

A-2 LOVIBOND TINTOMETER METHOD

A-2.1 The colour shall be determined by the method given in IS 1448 [P: 13] : 1960.

ANNEX B

[*Table* 1, *Sl No.* (vii) and *Clause* 7.1]

DETERMINATION OF BIODEGRADABILITY OF SULPHONATES

(SHAKE CULTURE METHOD)

B-0 OUTLINE OF THE METHOD

B-0.1 In this test, micro-organisms are inoculated into a flask that contains a chemically defined microbial growth medium (basal medium) and the surfactant to be tested. Aeration is accomplished by continuous shaking of the flask. Following two adoptive transfers, biodegradation is determined by measuring the reduction in surfactant content during the test period.

B-1 APPARATUS

B-1.1 Shaking Machine

A reciprocating shaker operating at about 128 strokes of 51 to 101.6 mm/min or a gyratory shaker operating at 225 to 250 rev/min with an amplitude of 25 to 51 mm should be used. Other shakers may be used if equivalent aeration can be demonstrated.

-2 REAGENTS

-2.1 Parity of Water

ither distilled or deionised water may be used. must be free of bacteriostatic materials. Water erived from steam condensate in many ceses ill contain amines which are inhibitory to icrobial growth.

-2.2 Basal Medium

he composition of the basal medium shall be s follows:

Ammonium chloride (NH_4Cl)	3.0 g
Potassium hydrogen phosphate (K_2HPO_4)	1.0 g
Magnesium sulphate heptahydrate ($MgSO_4 . 7H_2O$)	0.25 g
Potassium chloride (KCl)	0 25 g
Ferrous sulphate ($FeSO_4 . 7H_2O$)	0.002 g
Yeast extract	0.30 g
Water	1.01 g

-2.2.1 The basal medium may be prepared by equentially dissolving the dry ingredients in he water, or by adding stock solutions of the alts. The yeast extract shall be added in dry orm immediately before use, or alternatively, olutions containing yeast shall be sterilized and laintained sterile if held for more than 8 hours before beginning the test. The basal medium hall be dispensed into one of the following asks: 500 ml in a 1 litre flask, 1 000 ml in a -litre flask and 1 500 ml in a 4-litre flask. The -litre and 2-litre flasks are best suited for a yratory shake and the 4-litre flask for a -eciprocating shaker.

-2.3 Microbial Culture

-2.3.1 *Source*

he microbial inoculum may be obtained from ny of the following sources.

-2.3.1.1 Natural sources (soil, water, sewage, ctivated sludge, etc).

-2.3.1.2 Laboratory cultures (activated sludge, iver die-away, etc).

-2.3.2 *Maintenance of Culture*

f desired, the culture may be maintained as a hake flask culture by weekly transfers in the asal medium. For each weekly transfer use ml of 7-day culture for each 100 ml of fresh ledium.

B-3 PROCEDURE

Add 30 mg/1 surfactant to the flasks containing basal medium. If surfactant stock solutions are used, stability during storage shall be confirmed. Use one flask for each surfactant being tested and one blank flask containing all basal medium components but with no surfactant. Using the culture described in **B-2.3** inoculate the flasks. Use the same culture for all flasks including blank. Use 1 ml of inoculum for each 100 ml of basal medium in the flask. Place the flasks containing basal medium, surfactant and inoculum on a shaking machine that will produce acceptable aeration and mixing for biodegradation. Maintain the temperature of the flask contents at 25 ± 3°C. Prior to beginning the biodegradation test, make two 72-hours acclimation transfers from the flasks. To follow the course of biodegradation, remove samples from the shake flasks for analysis. Samples shall be taken during the 8-day test at zero time (immediately after inoculation and mixing of the flask contents) and on the seventh and eighth days. Samples at zero time of the two adaptive transfers are desirable to ensure proper initial concentration. Unless analyses are run immediately, the addition of 1 ml of formaldehyde/100 ml of sample shall be used for preservation of any sample (0 time or 7 or 8 days). When preservative is used, add to all samples including blank. Since the analytical result from the blank sample is used to correct the results from other flasks, use the same sample size (or dilution factor) for the blank as used for the other samples.

B-4 CALCULATION

Calculate net surfactant concentration by subtracting the analyzed blank values from the analyzed values for the other flasks. Calculate the percentage removal from the reduction in surfactant concentration as follows:

Percentage removal (day x)

$$= \left[\frac{(S_0 - B_0) - (S_r - B_z)}{(S_0 - B_0)} \right] \times 100$$

where

S_0 and S_r = analysis of test surfactant cultures, and

B_0 and B_z = analysis of blank cultures, on days 0 and x, respectively.

The result shall be calculated as the average of the seventh and eighth day percentage removals.

ANNEX C

[*Table* 1, *Sl No.* (ix) and *Clause* 7.1]

DETERMINATION OP MOLECULAR WEIGHT (GAS LIQUID CHROMATOGRAPHIC METHOD)

C-0 OUTLINE OF THE METHOD

C-0.1 The gas liquid chromatogram of the sample is recorded as described in **D-1** and assignments made as in **C-2.**

C-1 PROCEDURE

C-1.1 The linear alkyl benzene (LAB) sample is injected and the chromatogram is obtained as per the method described in **D-1**. Calculate the weight percent of all the linear alkyl benzene isomers; for example C_{10}, C_{11}, C_{12}... etc; after adjusting its area percent with response factors obtained by injecting the synthetic blend under identical conditions. Add up all the C_{10} isomers which will be the total C_{10}-LAB mass percent in the sample. Likewise calculate for all the carbon number LAB's such as C_{11}-LAB, C_{12}-LAB, etc. Calculate the total linear alkyl benzene content in the sample by adding up all weight percentages of C_{10}-LAB, C_{11}-LAB, etc.

C-2 CALCULATION

C-2.1 The percentage of different homolog LAB with respect to total linear alkyl benzene in the sample is calculated as follows:

$$X = \frac{Y}{Z} \times 100$$

where

X = weight percent of LAB-C_{10}, LAB-C_{11}, ... etc, with respect to LAB in the sample;

Y = weight percent of LAB-C_{10}, LAB-C_{11} . . . . etc, in the sample; and

Z = total linear alkyl benzene in the sample.

C-2.2 Average Molecular Weight

C-2.2.1 The average molecular weight of linear alkyl benzene is obtained by multiplying each percentage homolog LAB, as obtained from **C-2.1**, with its molecular weight and adding up all the values.

$$M = \frac{A \times C_{10} + B \times C_{11} + C \times C_{12} + D \times C_{13} + E \times C_{14} + \ldots\ldots}{100}$$

where

M = average molecular weight of linear alkyl benzene;

C_{10}, C_{11}...etc = weight percent of LAB-C_{10}, LAB-C_{11}, etc. . . . . ; and

A, B, C...etc = molecular weight of LAB-C_{10}, LAB-C_{11}, etc

ANNEX D

[*Table* 1, *Sl No.* (x) and *Clause* 7.1]

DETERMINATION OF PARAFFINS

D-0 OUTLINE OF THE METHOD

D-0.1 The sample to be analysed is injected into a gas chromatograph that is equipped with fused silica capillary column. A quantitative blend, containing known amount of normal paraffins in linear alkyl benzene (LAB) is chromatographed under identical conditions. The *calibration chromatogram* thus developed is used to ascertain the normal paraffins in the sample by comparing the retention of the normal paraffins to the observed retention times of the components obtained in the synthetic mixture. Quantitative results are obtained from the measured areas of the recorded peaks, adjusted for differences in response by the use of factors determined from the analysis of synthetic blend.

D-1 PROCEDURE

D-1.1 Inject C_{10} to C_{14} individual normal paraffins namely decane, undecane dodecane, tridecane and tetradecane into GC and find out the retention times of each paraffin. Prepare a synthetic blend containing close to 0.2 by mass of total normal paraffin in linear alkyl benzene. Now inject the synthetic blend and run the chromatogram. Identify the normal paraffin peaks from C_{10} to C_{14}. Calculate the area percent of individual normal paraffin and sum it up to get total area percent of the total normal paraffin in the blend. Calculate the area response factor as given in **D-2.1.**

D-1.2 Inject linear alkyl benzene sample and run the chromatogram. From the area percent of total normal paraffin calculate the weight percent as given in **D-2.2**.

D-2 CALCULATION

D-2.1 The area response factor is calculated as follows:

$$F = \frac{W}{A}$$

where

F = response factor;

W = weight percent of total normal paraffin in the blend; and

A = area percent of total normal paraffin in the blend;

D-2.2 The weight percent of totat normal paraffin in linear alkyl benzene as follows:

Percent total normal paraffin in linear alkyl benzene = $B \times F$

where

B = area percent of total normal paraffin obtained from the chromatogram; and

F = response factor of total normal paraffin.

ANNEX E

[*Table* 1, *Sl No.* (xi) and *Clause* 7.1]

DETERMINATION OF SULPHONABILITY

E 0 OUTLINE OF THE METHOD

E-0.1 A known mass of linear alkyl benzene of known molecular mass is sulphonated with 20 percent oleum. Knowing the mass of the product after sulphonation and its active matter content, the percent sulphonability is calculated.

E-1 APPARATUS

E-1.1 A 500-ml capacity 4-necked round bottom flask, with standard joints, is fitted with a mercury-sealed stirrer, thermometer (0-110°C) and a 100-ml, capacity addition funnel with pressure equalising side-arm (*see* Fig. 1). A guard-tube filled with granular, fused calcium chloride is inserted into the fourth neck of the flask.

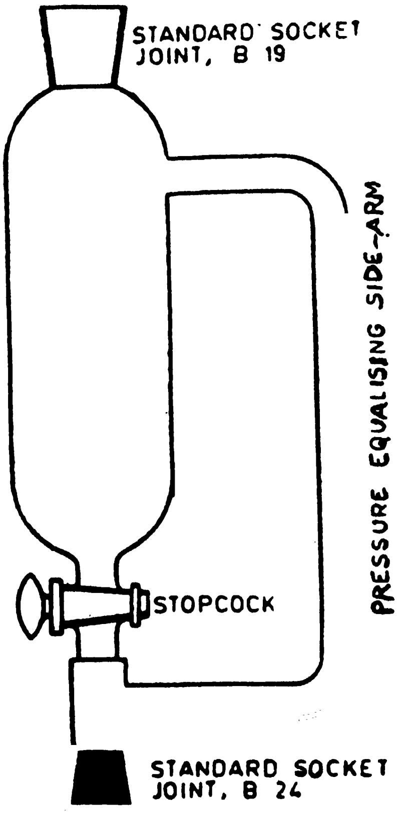
STANDARD SOCKET JOINT, B 19
PRESSURE EQUALISING SIDE-ARM
STOPCOCK
STANDARD SOCKET JOINT, B 24

FIG. 1 APPARATUS FOR DETERMINATION OF SULPHONABILITY

E-2 REAGENTS

E-2.1 20 Percent Oleum

E-2.2 Purified Hyamine 1622

Commercially-available, Hyamine 1622 is crystallised 6 to 7 times from chloroform-petroleum ether (60-80°). In the last crystallisation, it is dissolved in chloroform and filtered through filter paper to remove traces of dust and dirt. Then, the chloroform solution is concentrated on a hot-water bath and petroleum ether is added to the hot solution until it is slightly hazy. This solution is then cooled, scratching the sides with a clean glass rod. The separated crystals are filtered in a Buchner funnel under vacuum and the cake of crystals pressed dry with an inverted bottle stopper. The crystals are then scraped gently from the filter-paper into a glass dish and dried subsequently on a steam-bath. The melting point of the dried crystals is 163.5°C. (Literature, 164-166°C).

E-3 PROCEDURE

E-3.1 The empty 500-ml round bottomed flask, fitted with stirrer and thermometer (partly coming in contact with the reaction mixture) is weighed, to the second decimal 100.00 g of the linear alkyl benzene sample is now weighed into it. The mercury seal and calcium chloride guard tube are fitted. 120 g of 20 percent oleum is weighed into the addition funnel, which is stoppered at once and fitted to the 4-necked flask. The linear alkyl benzene is then cooled with stirring in a bath containing ice and water to 20°C. Dropwise addition of the 20 percent oleum is started. The reaction is exothermic, but, by adding more ice to the bath, reaction temperature is held below 25°C and a fairly fast rate of addition of oleum is maintained. After all the oleum is added, reaction mixture temparature is allowed to come up to 40°C, by replacing ice and water in the bath with warm water. The reaction mature is maintained at 40°C with stirring for 1 hour. The calcium chloride guard tube, additional funnel and mercury seal are removed. The outside of the flask is wiped dry and weighed, as before, with stirrer and thermometer, to the second decimal. The net weight of the contents after sulphonation is thus found out Its anionic active matter content is then found out as given in **A-4.2** of IS 8401 : 1977 using hyamine 1662 solution.

E-4 CALCULATION

$$\text{Percent sulphonability} = \frac{M_1 \times A \times M_2}{100(M_2 + 80)}$$

where

M_1 = nett mass of reaction mixture after sulphonation;

A = percent active matter of the sulphonated mixture; and

M_2 = average molecular mass of the sample.

ANNEX F

[*Table* 1, *Sl No.* (xii) and *Clause* 7.1]

DETERMINATION OF CHAIN LENGTH DISTRIBUTION

F-0 OUTLINE OF THE METHOD

F-0.1 The gas liquid chromatogram of the sample is taken and recorded as described in **D-1** and assignments made as in **C-1**. Thereafter, the chain length distribution of the sample is calculated as given in **C-2.1.**

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices:		Telephone
Central :	Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	311 01 31 331 13 75
Eastern :	1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola CALCUTTA 700054	37 86 62
Northern :	SCO 445-446, Sector 35-C, CHANDIGARH 160036	53 38 43
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